

## **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

## **Order Summary Report**

Order Number:	J13110223									
Project Name:										
Customer Name(s):	Bill Kennedy, Wayne Chapman									
Customer Address:	3195 Pine Hall Rd									
	Mailcode: Belews Steam Station									
	Belews Creek, NC 28012									
Lab Contact:	Jason C Perkins	Phone:	980-875-5348							
Report Authorized By: (Signature)		Dat	te:	12/6/2013						
(0.5	Jason C Perkins									

#### **Program Comments:**

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

#### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## Sample ID's & Descriptions:

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Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2013027767	BELEWS	13-Nov-13 7:30 AM	Matt C.	FGD Purge Eff
2013027768	BELEWS	13-Nov-13 7:45 AM	Matt C.	EQ Tank Eff
2013027769	BELEWS	13-Nov-13 7:50 AM	Matt C.	BIOREACTOR 1 INF
2013027770	BELEWS	13-Nov-13 7:55 AM	Matt C.	BioReactor 2 Inf
2013027771	BELEWS	13-Nov-13 8:00 AM	Matt C.	BioReactor 2 Eff
2013027773	BELEWS	13-Nov-13 8:30 AM	Matt C.	FILTER BLANK
2013027774	BELEWS	31-Oct-13 2:10 PM	D. Baker	METALS TRIP BLANK
7 Total Samples				

## **Technical Validation Review**

## **Checklist:**

COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures).	<b>✓</b> Yes	☐ No
All Results are less than the laboratory reporting limits.	Yes	<b>✓</b> No
All laboratory QA/QC requirements are acceptable.	<b>✓</b> Yes	☐ No

## **Report Sections Included:**

Reviewed By:

**DBA Account** 

✓ Job Summary Report	✓ Sub-contracted Laboratory Results
✓ Sample Identification	$\square$ Customer Specific Data Sheets, Reports, & Documentation
✓ Technical Validation of Data Package	☐ Customer Database Entries
✓ Analytical Laboratory Certificate of Analysis	✓ Chain of Custody
☐ Analytical Laboratory QC Report	✓ Electronic Data Deliverable (EDD) Sent Separatel

Date:

12/6/2013

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## Order # J13110223

Site: FGD Purge Eff Sample #: 2013027767

Collection Date: 13-Nov-13 7:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC								
Bromide	77	mg/L		5	50	EPA 300.0	11/22/2013 13:34	JAHERMA
Chloride	6700	mg/L		100	1000	EPA 300.0	11/22/2013 13:34	JAHERMA
MERCURY (COLD VAPOR) II	N WATER							
Mercury (Hg)	147	ug/L		5	100	EPA 245.1	11/26/2013 12:42	DKJOHN2
TOTAL RECOVERABLE MET	ALS BY ICP							
Boron (B)	225	mg/L		0.5	10	EPA 200.7	11/19/2013 16:14	SLS2560
DISSOLVED METALS BY ICE	P-MS							
Selenium (Se)	933	ug/L		10	10	EPA 200.8	11/26/2013 14:11	DJSULL1
TOTAL RECOVERABLE MET	ALS BY ICP-MS							
Arsenic (As)	193	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Chromium (Cr)	297	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Copper (Cu)	122	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Nickel (Ni)	240	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Selenium (Se)	2830	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Thallium (TI)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Vanadium (V)	202	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
Zinc (Zn)	233	ug/L		10	10	EPA 200.8	12/04/2013 13:33	DJSULL1
SELENIUM SPECIATION - (A	nalysis Performed	by Applied	Speciation a	nd Cons	ulting, LLC	<u>s)</u>		
Vendor Parameter	Complete					Vendor Method		V_AS&C
TOTAL DISSOLVED SOLIDS								
TDS	23000	mg/L		250	1	SM2540C	11/20/2013 14:13	DSBAKE1
TOTAL SUSPENDED SOLIDS	<u>5</u>							
TSS	3000	mg/L		250	1	SM2540D	11/15/2013 13:30	TJA7067
0"						<b>2</b> 1 " 00	40007700	

Site: EQ Tank Eff Sample #: 2013027768

Collection Date: 13-Nov-13 7:45 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst		
MERCURY (COLD VAPOR) IN WATER										
Mercury (Hg)	157	ug/L		2.5	50	EPA 245.1	11/26/2013 12:45	DKJOHN2		
TOTAL RECOVERABLE METALS BY ICP										
Boron (B)	236	mg/L		0.5	10	EPA 200.7	11/19/2013 16:18	SLS2560		

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## Order # J13110223

Site: EQ Tank Eff Sample #: 2013027768

Collection Date: 13-Nov-13 7:45 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst		
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	486	ug/L		10	10	EPA 200.8	11/26/2013 14:14	DJSULL1		
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	230	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Chromium (Cr)	366	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Copper (Cu)	153	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Nickel (Ni)	263	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Selenium (Se)	3290	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Thallium (TI)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Vanadium (V)	271	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		
Zinc (Zn)	281	ug/L		10	10	EPA 200.8	12/04/2013 13:36	DJSULL1		

Site: BIOREACTOR 1 INF Sample #: 2013027769

Collection Date: 13-Nov-13 7:50 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst		
NITRITE + NITRATE (COLORIME	TRIC)									
Nitrite + Nitrate (Colorimetric)	7.7	mg-N/L		0.25	25	EPA 353.2	11/15/2013 11:46	BGN9034		
INORGANIC IONS BY IC										
Bromide	75	mg/L		5	50	EPA 300.0	11/22/2013 13:53	JAHERMA		
Chloride	6800	mg/L		100	1000	EPA 300.0	11/22/2013 13:53	JAHERMA		
Mercury by EPA 200.8 - (Analysis	s Performed by	Applied Sp	peciation and	Consult	ing, LLC)					
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C		
TOTAL RECOVERABLE METALS	BY ICP									
Boron (B)	213	mg/L		0.5	10	EPA 200.7	11/19/2013 16:22	SLS2560		
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	<u>2</u> 549	ug/L		10	10	EPA 200.8	11/26/2013 14:17	DJSULL1		

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## Order # J13110223

Site: BIOREACTOR 1 INF Sample #: 2013027769

Collection Date: 13-Nov-13 7:50 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	BY ICP-MS							
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Nickel (Ni)	12.6	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Selenium (Se)	561	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Thallium (TI)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Vanadium (V)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:40	DJSULL1
SELENIUM SPECIATION - (Analys	sis Performed b	y Applied	Speciation a	nd Consu	ulting, LLC	<u>C)</u>		
Vendor Parameter	Complete					Vendor Method		V_AS&C
TOTAL DISSOLVED SOLIDS								
TDS	15000	mg/L		25	1	SM2540C	11/20/2013 14:13	DSBAKE1
TOTAL SUSPENDED SOLIDS								
TSS	9.0	mg/L		5	1	SM2540D	11/15/2013 13:30	TJA7067

Site: BioReactor 2 Inf Sample #: 2013027770

Collection Date: 13-Nov-13 7:55 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst		
Mercury by EPA 200.8 - (Analysis	Performed by	Applied Sp	eciation and	Consulti	ng, LLC)					
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C		
TOTAL RECOVERABLE METALS BY ICP										
Boron (B)	209	mg/L		0.5	10	EPA 200.7	11/19/2013 16:26	SLS2560		
TOTAL RECOVERABLE METALS BY ICP-MS										
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Nickel (Ni)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Selenium (Se)	20.4	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Thallium (TI)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Vanadium (V)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	12/04/2013 13:43	DJSULL1		

DJSULL1

DJSULL1

DJSULL1

DJSULL1

12/04/2013 13:47

12/04/2013 13:47

12/04/2013 13:47

12/04/2013 13:47

## **Certificate of Laboratory Analysis**

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#### Order # J13110223

Site: BioReactor 2 Inf Sample #: 2013027770

Collection Date: 13-Nov-13 7:55 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

Site: BioReactor 2 Eff
Collection Date: 13-Nov-13 8:00 AM

Result Units Qualifiers RDL DF Method Analysis Date/Time Analyst

Sample #: 2013027771

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst		
NITRITE + NITRATE (COLORIMETE	RIC)									
Nitrite + Nitrate (Colorimetric)	0.016	mg-N/L		0.01	1	EPA 353.2	11/15/2013 11:47	BGN9034		
INORGANIC IONS BY IC										
Bromide	77	mg/L		5	50	EPA 300.0	11/22/2013 14:12	JAHERMA		
Chloride	6600	mg/L		100	1000	EPA 300.0	11/22/2013 14:12	JAHERMA		
Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)										
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C		
TOTAL RECOVERABLE METALS E	BY ICP									
Boron (B)	205	mg/L		0.5	10	EPA 200.7	11/19/2013 16:30	SLS2560		
TOTAL RECOVERABLE METALS E	BY ICP-MS									
Arsenic (As)	< 5	ug/L		5	5	EPA 200.8	12/04/2013 13:47	DJSULL1		
Cadmium (Cd)	< 5	ug/L		5	5	EPA 200.8	12/04/2013 13:47	DJSULL1		
Chromium (Cr)	< 5	ug/L		5	5	EPA 200.8	12/04/2013 13:47	DJSULL1		
Copper (Cu)	< 5	ug/L		5	5	EPA 200.8	12/04/2013 13:47	DJSULL1		
Nickel (Ni)	< 5	ug/L		5	5	EPA 200.8	12/04/2013 13:47	DJSULL1		
Selenium (Se)	6.51	ug/L		5	5	EPA 200.8	12/04/2013 13:47	DJSULL1		

### SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)

< 5

< 5

< 5

< 5

Silver (Ag)

Zinc (Zn)

Thallium (TI)

Vanadium (V)

Vendor Parameter Complete Vendor Method V\_AS&C

5

5

5

5

5

5

EPA 200.8

EPA 200.8

EPA 200.8

EPA 200.8

Site: FILTER BLANK Sample #: 2013027773

Collection Date: 13-Nov-13 8:30 AM Matrix: OTHER

ug/L

ug/L

ug/L

ug/L

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS								
Selenium (Se)	1.18	ug/L	B2	1	1	EPA 200.8	11/26/2013 13:56	DJSULL1

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### Order # J13110223

Site: METALS TRIP BLANK Sample #: 2013027774

Collection Date: 31-Oct-13 2:10 PM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS B	Y ICP							
Boron (B)	< 0.05	mg/L		0.05	1	EPA 200.7	11/19/2013 16:06	SLS2560
TOTAL RECOVERABLE METALS B	Y ICP-MS							
Arsenic (As)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Cadmium (Cd)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Chromium (Cr)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Copper (Cu)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Nickel (Ni)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Selenium (Se)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Silver (Ag)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Thallium (TI)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Vanadium (V)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1
Zinc (Zn)	< 1	ug/L		1	1	EPA 200.8	12/04/2013 13:26	DJSULL1

### Qualifiers:

**B2** Target analyte was detected in blank(s) at a concentration greater than ½ the reporting limit but less than the reporting limit. Analyte concentration in sample is valid and may be used for compliance purposes.



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

December 5, 2013

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling) (LIMS# J13110223)

Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for total mercury, hexavalent chromium, and selenium speciation analyses on November 14, 2013. The samples were received in a sealed cooler at -0.3°C on November 15, 2013. Mercury quantitation was performed via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS). Hexavalent chromium analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Any issues associated with the analyses are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Jeremy Maute Project Coordinator

Applied Speciation and Consulting, LLC

## Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078

Project: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling) (LIMS# J13110223)

December 5, 2013

#### 1. Sample Reception

Three (3) aqueous samples were submitted for hexavalent chromium and selenium speciation analyses on November 14, 2013. Three (3) additional samples were submitted for total mercury quantitation. All samples were received in acceptable condition on November 15, 2013 in a sealed container at -0.3°C.

All samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. The 40mL borosilicate glass vials submitted for total mercury were preserved with bromine monochloride (BrCl) solution. The resulting samples were stored in a secure polyethylene container, known to be free from trace metals contamination, until the analyses could be performed.

An aliquot of each sample submitted for hexavalent chromium analysis was filtered (0.45um) and stored in a secure refrigerator maintained at a temperature of 4°C, until the analyses could be performed.

An aliquot of each sample requiring selenium speciation evaluation was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

#### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Total Mercury Quantitation by CV-ICP-MS</u> All samples and preparation blanks for total mercury quantitation were preserved with 2% (v/v) BrCl. The resulting samples were analyzed for mercury via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS).

<u>Hexavalent Chromium Analysis by IC-ICP-DRC-MS</u> Prior to analysis, all samples were injected directly into autosampler vials. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

<u>Selenium Speciation Analysis by IC-ICP-CRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45μm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

## 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

<u>Total Mercury Quantitation by CV-ICP-MS</u> The sample fractions for total mercury quantitation were analyzed by cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS) on November 18, 2013. Aliquots of each sample are reacted with a reductant in-line and transported to a gas-liquid separator. The volatile elemental mercury that is formed is then swept by a stream of argon gas into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and separated on the basis of their mass-to-charge ratio (m/z) by a mass spectrometer. A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

<u>Hexavalent Chromium Analysis by IC-ICP-DRC-MS</u> Each sample for hexavalent chromium analysis was analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on December 5, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The

eluting chromium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

<u>Selenium Speciation Analysis by IC-ICP-CRC-MS</u> Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on November 19, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

#### 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL values for methylseleninic acid and selenomethionine are calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

The eMDL values for hexavalent chromium and mercury are calculated using the standard deviation of the preparation blanks preserved and analyzed concurrently with the submitted samples.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Jeremy Maute

Project Coordinator

Applied Speciation and Consulting, LLC

## Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy Project Name: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling) Contact: Jay Perkins LIMS #J13110223

Date: December 5, 2013
Report Generated by: Jeremy Maute
Applied Speciation and Consulting, LLC

### Sample Results

								Unknown Se
Sample ID	Total Hg	Cr(VI)	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	NR	ND (< 1.1)	1450	23.3	3.0	ND (< 1.7)	ND (< 1.7)	3.6 (1)
BioReactor 1 Inf	0.0703	ND (< 1.1)	538	21.9	5.29	3.29	ND (< 0.43)	1.77 (1)
BioReactor 2 Inf	0.0124	NR	NR	NR	NR	NR	NR	NR
BioReactor 2 Eff	0.0054	ND (< 1.1)	ND (< 0.47)	ND (< 0.30)	ND (< 0.53)	ND (< 0.43)	ND (< 0.43)	0 (0)

All results reflect the applied dilution and are reported in µg/L

NR = Analysis not requested

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

# Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy Project Name: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling) Contact: Jay Perkins LIMS #J13110223

Date: December 5, 2013
Report Generated by: Jeremy Maute
Applied Speciation and Consulting, LLC

## **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 5x	eMDL 250x	eMDL 1000x
Hg	-0.0004	-0.0003	-0.0001	-0.0002	-0.0003	0.0001	0.0001	0.0004	-	-
Cr(VI)	-0.4	-1.0	-0.2	-0.2	-0.5	0.4	0.001	-	-	1.1
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.47	1.9
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.001	-	0.30	1.2
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.53	2.1
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.43	1.7
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.43	1.7

eMDL = Estimated Method Detection Limit

## **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Hg	NIST 1641d	1568	1626	103.7
Cr(VI)	LCS	1.000	1.161	116.1
Se(IV)	LCS	9.57	9.28	96.9
Se(VI)	LCS	9.48	9.22	97.3
SeCN	LCS	8.92	8.55	95.9
MeSe(IV)	LCS	6.47	6.23	96.3
SeMe	LCS	9.32	8.84	94.8

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy Project Name: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling) Contact: Jay Perkins LIMS #J13110223

Date: December 5, 2013
Report Generated by: Jeremy Maute
Applied Speciation and Consulting, LLC

#### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Hg	BioReactor 2 Eff	0.0054	0.0053	0.0054	1.9
Cr(VI)	Batch QC	ND (< 1.1)	ND (< 1.1)	NC	NC
Se(IV)	Batch QC	ND (< 0.47)	ND (< 0.47)	NC	NC
Se(VI)	Batch QC	ND (< 0.30)	ND (< 0.30)	NC	NC
SeCN	Batch QC	ND (< 0.53)	ND (< 0.53)	NC	NC
MeSe(IV)	Batch QC	ND (< 0.43)	ND (< 0.43)	NC	NC
SeMe	Batch QC	ND (< 0.43)	ND (< 0.43)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

## **Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Hg	BioReactor 2 Eff	2.000	2.106	105.0	2.000	2.119	105.7	0.6
Cr(VI)	Batch QC	1000	860.1	86.0	1000	845.7	84.6	1.7
Se(IV)	Batch QC	1390	1336	96.1	1390	1344	96.7	0.6
Se(VI)	Batch QC	1261	1176	93.3	1261	1190	94.3	1.1
SeCN	Batch QC	1144	1026	89.7	1144	1043	91.2	1.7

#### <sup>22</sup>Requested Turnaround Page 17 of 18 ORIGINAL to LAB. \*Other \* Add. Cost Will Apply COPY to CLIENT 19 Page 1 of 2 DISTRIBUTION cottle back into both baggies) Filtering of the Se is performed in the field please provide a filter blank too. (Important to place filled Cr, Se, speciation - vendor 14 Days \*7 Days ·48 Hr Nitrate **Drinking Water** 19 200.8 (V\_AS&C) UST SAMPLE PROGRAM Water **RCRA Waste** 3,4 Se (IMS), filtered CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM 43.4 \*\* \*\* \*\* Netals\* + Hg\*\* Samples Originating From Br, CI (Dionex) Analytical Laboratory Use Only TDS, TSS -Cooler Temp (C) <sup>15</sup>Preserv.:1=HCL 2=H<sub>2</sub>SO<sub>4</sub> 3=HN<del>O</del><sub>9</sub> Required Grab 5=None easylses Comp. appropriate non-shaded areas. Customer to complete all Signature Defline Mat Could Offline Matt Com 1/13 0755 MAY CO. Mast Com Date & Time 1/13 0750 Mat PH ON=" PO #650910 1311022 0230 346 0830 0800 Time 0 31 13 1410 1 10) Seal/Lock Opened 8)Accepted By: As, Cd, Cr, Cu, Ni, Se, Ag, Zn, V, TI by TRM/IMS 11/13 11/13 Date 11/13 <sup>13</sup>Sample Description or ID **Duke Energy Analytical Laboratory** 1830000 Mail Code MGO3A2 (Building 7405) Mail Code: 10)Reso. Center: 13339 Hagers Ferry Rd Huntersville, N. C. 28078 (704) 875-5245 BioReactor 2 Inf BioReactor 2 Eff BioReactor 1 Inf FGD Purge Eff Metals Trip Blk 2)Phone No EQ Tank Eff. mF Pilot Eff Bio Pilot Eff Fax: (704) 875-4349 1)Fax No: Filter BIK 11/13/13 Date/Time Date/Time SFHDW1205 Fontier Pilot (Belews Creek) Bill Kennedy, Wayne Chapman 3500 9)Res. Type 6)Process: Se Speciation Bottle \* B by TRM/ICP 20006 FHGO Relinquished By 1)Seal/Locked By Seal/Locked By LAB USE ONLY ()Business Unit 12178 177 Project Name 020250 "Lab ID 8)Oper. Unit: 307

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